Vacuum 85 (2011) 772–775

Contents lists available at ScienceDirect

Vacuum

journal homepage: www.elsevier.com/locate/vacuum

The properties of aluminium coating on sintered NdFeB by DC magnetron sputtering

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ARTICLE INFO

Article history:
Received 2 September 2010
Received in revised form
17 November 2010
Accepted 19 November 2010

Keywords:
NdFeB
Aluminium coating
Magnetron sputtering
Corrosion resistance
Adhesive strength
Magnetic properties

ABSTRACT

Pure aluminium coating was deposited on sintered NdFeB magnets by direct current (DC) magnetron sputtering to improve the corrosion resistance. The corrosion behaviour of sintered NdFeB coated with aluminium was characterized by potentiodynamic polarization measurement. The adhesive strength between the aluminium coating and the sintered NdFeB was characterised by vertical tensile test and high-low temperature shock test. The influence of the coating on the magnetic properties of the sintered NdFeB was also characterised. It was found that the aluminium coating can improve the corrosion resistance of the sintered NdFeB evidently. The aluminium coating was well adhesive with the substrate and did not deteriorate the magnetic properties of the sintered NdFeB magnets. These two characters may overcome the disadvantages of NiCuNi coating which is usually electroplated on NdFeB in industry.

1. Introduction

Sintered NdFeB magnets have been widely used for their remarkable magnetic properties [1]. However, the poor corrosion resistance of the sintered NdFeB magnets in various environments hinders their further application [2–5]. Their poor corrosion resistance is due to their microstructure which is comprised by the matrix phase, which is ferromagnetic tetragonal compound $Nd_2Fe_{14}B$, the Nd-rich phase Nd_4Fe, and the B-rich phase $Nd_{1+\varepsilon}Fe_4B_4$. Galvanic corrosion is easy to happen between the electrochemically active Nd-rich phase and the matrix phase [5,6].

To improve the chemical stability of the sintered NdFeB magnets, many attempts have been employed, e.g. alloy additions [7,8] and surface coatings [9,10]. At industry level, electroplating is a widely used method. However, this method not only always involves in the problem of environmental concerns, but also deteriorates the magnetic properties of the magnets [11,12]. Moreover, for their inferior adhesion, the electroplated coatings on sintered NdFeB are susceptible to fail in the condition of frequent high-low temperature shock.

Physical Vapour Deposition (PVD) is a kind of dry technology without liquid waste pollution comparing with the electroplating. It can provide coatings to the substrate with better adhesion which is critical to the corrosion resistance in the condition of frequent high-low temperature shock. Meanwhile, the efficiency of PVD has been enhanced in industry in recent years [11]. In some studies [13–15], Al, Al/Ti and Al/Cr coatings by PVD have been applied to the corrosion resistance of magnesium alloys, aluminium alloys and steels, respectively. For the sintered NdFeB products, aluminium coating is one of the latent candidates for protection due to its friendly price and good corrosion resistance in aggressive media with pH between 4 and 9 [16–18]. Aluminium coatings by thermal evaporation have been applied to improve the corrosion resistance of sintered NdFeB [19–21].

However, the comparison of the properties of PVD coatings and electroplated coatings on the sintered NdFeB magnets has not been well studied. This work focuses on the properties of the sintered NdFeB coated with aluminium by DC magnetron sputtering (Al/NdFeB). The magnetic properties of the Al/NdFeB and the sintered NdFeB coated with NiCuNi triple layers by electroplating (NiCuNi/NdFeB) were also compared.

2. Experimental details

The sintered NdFeB specimens (banded 35H, Yunshen Co. Ltd, in demagnetisation state) with a size of 20 mm × 10 mm × 5 mm were ground and polished to a mirror surface and then ultrasonically cleaned in acetone followed by alcohol. The specimens were used for the tests of scanning electron microscopy (SEM, S-4800, Hitachi),

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doi:10.1016/j.vacuum.2010.11.016

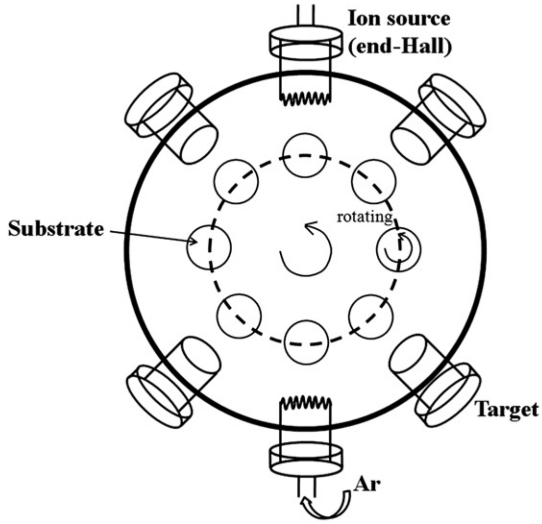

Fig. 1. The scheme of the magnetron sputtering apparatus designed for magnets protection.

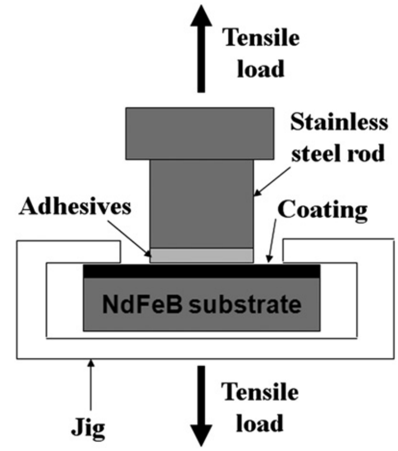

Fig. 2. The scheme of vertical tensile test mount.

glancing angle X-ray diffraction (GXRD, D8, Bruker) and potentiodynamic polarization. For comparison, as treated in industry by electroplating, other part of sintered NdFeB specimens were cleaned by a weak alkaline solution followed by a 3% HNO_3 solution for the tests of adhesive strength (with a size of 25 mm × 25 mm × 3 mm) and magnetic properties (with a size of ϕ 10 mm × 10 mm).

Deposition of aluminium coating was carried out in a magnetron sputtering apparatus designed for magnets protection, which can deposit homogeneous coatings onto all surfaces of the specimens with batch treatments. The scheme of the apparatus was shown in Fig. 1. The chamber was evacuated to a base pressure of 5×10^{-4} Pa. Pure Ar (99.999%) was introduced at a constant flow rate of 40 sccm, for maintaining the desired sputtering pressure of 0.5 Pa. Before deposition, the specimens were cleaned by Ar^+ ion beams provided by two end-Hall ion guns with a energy 150 eV in different time to enhance the adhesive strength [17]. In addition, the Al/NdFeB without pre-cleaning by the end-Hall ion guns was made as references. The aluminium coating was then prepared by DC magnetron sputtering from four Al targets (99.999%). During deposition, the magnets ran with both revolution and rotation with its top surface and side surfaces facing to the targets and the ion guns in a calculated angle. The thickness of the coatings on the side surfaces were half of that on the top surface. And then, the magnets were turned over to deposit coatings on the bottom and meanwhile the side surfaces. The thickness of the coating which was measured by a surface profilometer (Alpha-Step, IQ) employing a step formed by a shadow mask was approximately 5 μm.

For comparison, NiCuNi triple layers coating which are usually used in industry were electroplated by barrel plating on sintered NdFeB specimens in sequence. The thicknesses of Ni (inner layer), Cu and Ni layers were approximately 8 μm, 8 μm and 4 μm, respectively.

The corrosion behaviour of the Al/NdFeB was investigated by potentiodynamic polarization in 3.5 wt. % NaCl solutions at 25 ± 3 °C using an Autolab potentiostat (PGSTAT302, Ecochimie). A conventional three-electrode cell was used with an Ag/AgCl (saturated KCl) as the reference electrode and a platinum sheet (20 mm × 10 mm) as the auxiliary electrode. The specimens were kept in solutions for 1 h before measurement for the stabilisation of the stationary potential.

The adhesive strength between the coating and the substrate was characterised by two different methods. The one method is vertical tensile test in a universal testing machine (CMT 5105). The schematic diagram of the test mount was shown in Fig. 2. A stainless steel rod with 18 mm diameter was glued onto the coating followed airing at room temperature over 72 h, and then a groove was made surrounding the steel rod to cut the contacted area off from other area. In testing, with increasing tensile load, the load-displacement curve was recorded until the coating was stripped out from the substrate NdFeB. The adhesive strength is derived from $S = L_{max}/A$, where S is the adhesive strength (unit: Pa), L_{max} is the maximum load value (unit: N). A is the contacted area between the stainless steel rod and the coating (unit: m^2).

The other method for adhesive strength test is to simulate the frequent high-low temperature shock environment. In testing, a bath of sand (diameter < 0.1 mm) which was heated up to 200 °C and a jar of liquid nitrogen (−196 °C) were employed as the sources for temperature shock. The Al/NdFeB specimens were transferred between the sand bath and the liquid nitrogen jar.

The influences of the aluminium coating and the NiCuNi coating on the magnetic properties of the sintered NdFeB were investigated by a NIM-500 hysteresgraph at 25 ± 1 °C. There were ten specimens for each condition. The uncoated specimens and the coated specimens were one to one correspondence.

3. Results and discussion

3.1. Characterisation of aluminium coating

Fig. 3 shows the surface and the cross-section micrographs of the aluminium coating. In Fig. 3a, many particles range from hundreds of nanometres to more than 1 μm. It is found that the aluminium coating exhibits a columnar structure in Fig. 3b. The GXRD pattern of the aluminium coating on the NdFeB specimen is presented in Fig. 4. It shows excellent crystallinity.

3.2. Potentiodynamic polarization measurement

Fig. 5 shows the potentiodynamic polarization curves of two different specimens: (a) the Al/NdFeB, and (b) the sintered NdFeB. The anodic curve of the Al/NdFeB exhibits a passive-like anodic behaviour as the potential increases to a value of −0.73 V (Ag/AgCl). It turns to stable pitting corrosion when the potential exceeds its pitting potential (−0.68 V (Ag/AgCl)) [22]. The broad passive region which may result from the formation of oxide film on the aluminium is beneficial to the protection of the substrate NdFeB. The corrosion current density i_{corr} were calculated by GPES software near zero overall current. It is found that the corrosion current